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EFFECT OF WASHING AND SOLAR DRYING ON THE THERMOCHEMICAL PROFILE OF *Sargassum* spp.

Pre-treatment effects on *Sargassum* spp. thermochemical profile

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Keywords: pelagic macroalgae, morphotype composition, coastal resources, waste valorization, energy recovery.

ABSTRACT

The massive influx of *Sargassum* along Caribbean coastlines presents both an environmental challenge and an opportunity for biomass valorization. This study evaluated pretreatment consisting of washing and solar drying to adapt *Sargassum* biomass for thermochemical recovery. Biomass was collected from three coastal sites in the Mexican Caribbean, with an average morphotype composition of *Sargassum natans III* (88.3%), *Sargassum fluitans I* (10%), and *Sargassum natans VIII* (1.6%). Samples were divided into Control (untreated) and Wash-dry (pretreated) groups and analyzed through Fourier transform infrared (FTIR) spectroscopy, proximate analysis, and ultimate analysis. Statistical evaluation revealed that prior segregation by morphotype or morphological section does not significantly influence biomass composition or higher heating value (~14 MJ/kg), supporting large-scale processing strategies. FTIR spectra showed that both groups retained key functional groups such as hydroxyl, carbonyl, and polysaccharide-associated vibrations, indicating biochemical integrity. The intensity of the signals associated with marine salts and sand decreased in the Wash-dry sample, confirming effective removal of surface-bound inorganic matter. Proximate analysis revealed a reduction in both moisture content (from 57.9 to 18.1%) and fixed carbon content (from 3.5 to 0.9%). Volatile matter increased from 20.7 to 59.4%, enhancing the potential of *Sargassum* for oil and gas production. Ultimate analysis revealed statistically similar C, H, N, S, and O contents after pretreatment, confirming preservation of the internal chemical structure. These findings demonstrate that the Wash-dry protocol is a selective and non-destructive strategy that enhances *Sargassum* biomass quality, potentially enabling a less energy-intensive pathway for its thermochemical valorization.

Palabras clave: macroalga pelágica, composición morfotípica, recursos costeros, valorización de residuos, valorización energética.

RESUMEN

El masivo arribo de sargazo a lo largo de las costas del Caribe representa tanto un desafío ambiental como una oportunidad para la valorización de biomasas. Este estudio evaluó un pretratamiento basado en el lavado y el secado solar para adaptar la biomasa de sargazo a procesos de valorización termoquímica. La biomasa fue recolectada en tres sitios costeros del Caribe mexicano, con una composición morfotípica promedio de *Sargassum natans* III (88.3 %), *Sargassum fluitans* I (10 %) y *Sargassum natans* VIII (1.6 %). Las muestras se dividieron en dos grupos: Control (sin tratamiento) y lavado-secado (pretratado) y se analizaron mediante espectroscopia infrarroja por transformada de Fourier (FTIR), análisis proximal y análisis elemental. La evaluación estadística reveló que la segregación previa por morfotipo o sección morfológica no influye significativamente en la composición de la biomasa ni en su poder calorífico superior (~14 MJ/kg), lo que respalda estrategias de procesamiento a gran escala. Los espectros FTIR mostraron que ambas muestras conservaron grupos funcionales clave, como hidroxilos y carbonilos, así como vibraciones asociadas a polisacáridos, lo que indica integridad bioquímica. La intensidad de las señales asociadas a sales marinas y arena disminuyó en la muestra Lavado-secado, lo que confirma la eliminación efectiva de materia inorgánica superficial. El análisis proximal reveló una reducción en el contenido de humedad (de 57.9 a 18.1 %) y carbón fijo (de 3.5 a 0.9 %). El material volátil aumentó de 20.7 a 59.4 %, lo que mejora el potencial del sargazo para la producción de aceite y gas. El análisis elemental mostró contenidos estadísticamente similares de C, H, N, S y O tras el pretratamiento, confirmando la preservación de la estructura química interna. Estos hallazgos demuestran que el protocolo de lavado-secado es una estrategia selectiva y no destructiva que mejora la calidad de la biomasa de sargazo y podría permitir una ruta de valorización termoquímica menos intensiva en energía.

INTRODUCTION

Sargassum (class: Phaeophyceae, order: Fucales, family: Sargassaceae) are pelagic macroalgae that proliferate across the equatorial Atlantic and arrive in massive quantities along Caribbean coastlines, including the Mexican coasts (Putman et al. 2023). These influxes have caused significant ecological disruptions, particularly affecting marine species such as neritic fish and crustaceans due to the decomposition of *Sargassum* along the shore (Vázquez-Delfín et al. 2024). For instance, in February 2022, approximately 97 000 t of *Sargassum* were recorded (Rodríguez-Martínez et al. 2023). The collected biomass is often discarded in nearby areas or in landfills lacking the infrastructure to manage such volumes, leading to ecological imbalances and reduced landfill lifespans (Rodríguez-Martínez et al. 2022).

Given its abundance and biochemical composition, *Sargassum* biomass is considered a promising feedstock for industrial by-product generation (Fidai et al. 2024). It contains cellulose, hemicellulose, and lignin-rich compounds that serve as precursors for high-value by-products (Stiger-Pouvreau et al. 2023). These by-products, which may occur in solid, condensable, or non-condensable forms, can be obtained through biological methods (e.g., fermentation, anaerobic digestion) or thermochemical processes (e.g., calcination, gasification, pyrolysis). However, the presence of cytotoxic metals such as arsenic, cadmium, lead, and boron limits its applicability in food-related processes and inhibits microbial activity in biological recovery pathways, such as anaerobic digestion (Devault et al. 2021). Moreover, the high content of insoluble fiber, salts, and polyphenols in *Sargassum* hinders biotransformation, making anaerobic digestion an inefficient valorization route (Davis et al. 2021).

In contrast, thermochemical valorization of *Sargassum* offers a robust alternative by employing high temperatures to decompose insoluble fibers, such as lignin, cellulose, and hemicellulose into solid residues, condensable oils, and non-condensable gases (Sanabria-Pérez et al. 2025). These by-products derived from macroalgae are highly valued in industries such as energy, pharmaceuticals, and catalysis (Alam et al. 2024). Despite extensive documentation on the thermochemical conversion of macroalgae, its application to collected *Sargassum* remains limited. Pyrolysis of *Sargassum* yields a high proportion of solid residue (> 39%), indicating a conversion rate below 60% (Davis et al. 2021). This inefficiency is largely attributed to its high moisture content (~70%), which requires substantial thermal energy, and to its elevated ash content (10 to 44%), which inhibits its reaction kinetics and reduces gas and oil yields (Tobío-Pérez et al. 2022).

Studies have demonstrated that washing and drying pretreatments significantly enhance the thermochemical valorization of marine biomass. It has been reported that applying a simple pre-treatment of freshwater rinsing and drying to beach wrack (seagrass or seaweed biomass), promoted a reduction in its ash content from 35.2 to 18.6%, while increasing the high heating value (HHV) from 8.4 to 13.2 MJ/kg, facilitating its conversion into biochar and syngas through pyrolysis (Rudovica et al. 2021). Another study found that desalination pretreatment removed up to 70% of surface-bound salts and reduced total inorganic content by 40%, improving reaction kinetics and reducing slag formation during gasification (Datta et al. 2024). Additionally, solar drying of *Sargassum fluitans*, *Ulva lactuca*, and *Chaetomorpha* cf. *gracilis* was also evaluated, achieving moisture loss peaks of 80% within 45 min and fitting the drying kinetics to a polynomial model with a determination coefficient of 99.13%, confirming the reliability of this method for biomass conditioning (Sánchez-Borroto et al. 2024). These findings support the strategic use of pretreatment protocols to optimize marine biomass for energy recovery. Despite its abundance, driven by the recurrent influx of *Sargassum* to Caribbean coastlines, and its compositional suitability, this biomass remains underutilized in this context, highlighting the relevance of this study in bridging that gap.

To improve the physical and chemical properties of collected *Sargassum* and enhance its thermochemical conversion potential, this study aimed to explore a pretreatment strategy, based on washing and solar drying, to adapt *Sargassum* biomass for thermochemical valorization. The proposed method aims to reduce moisture, eliminate surface-bound inorganic contaminants, and increase the availability of volatile matter, thereby optimizing the production of bio-oil and biogas. By refining *Sargassum* through a simple and scalable approach, this research offers a pathway to transform a burden into a resource for coastal communities.

MATERIALS AND METHODS

Collection, taxonomic analysis, and pretreatment of *Sargassum*

Figure 1 illustrates the three *Sargassum* biomass collection sites located on the coast of Quintana Roo, Mexico. The collection procedure involved using a calibrated tape to measure a 100 m transect in the intertidal zone, with biomass bundles collected at 10 m intervals. The collected *Sargassum* was washed with seawater and left to rest in the shade on a net (3 mm mesh size) for 4 h to maintain natural arrival conditions. In-situ taxonomic analysis was conducted on the collected *Sargassum*, involving the segregation and identification of species and morphotypes based on taxonomic guides (Siuda et al. 2024). Additionally, the wet mass content was evaluated using a spring balance.

Table I summarizes the pretreatment protocols applied for the analysis of *Sargassum* biomass. The Control condition refers to biomass collected directly from the shoreline at the three sampling sites, as shown in **figure 1**, under the specific temporal, thermal, and hygrometric conditions prevailing at each location. The Wash-dry pretreatment was designed to improve the physical and chemical properties of the biomass through a standardized laboratory procedure. This involved rinsing the *Sargassum* with distilled water until chloride ions were no longer detectable, followed by solar drying over three consecutive days. The solar drying protocol was adapted from the methodology proposed by Sánchez-Borroto et al. (2024), incorporating adjustments to accommodate local environmental conditions and the specific aims of the present study. This approach was deliberately chosen to emulate, as closely as possible, the natural exposure conditions experienced by *Sargassum* during its deposition and accumulation along coastal environments, while minimizing the energy and financial costs associated with conventional drying technologies. The drying protocol was implemented outdoors under controlled exposure, with continuous

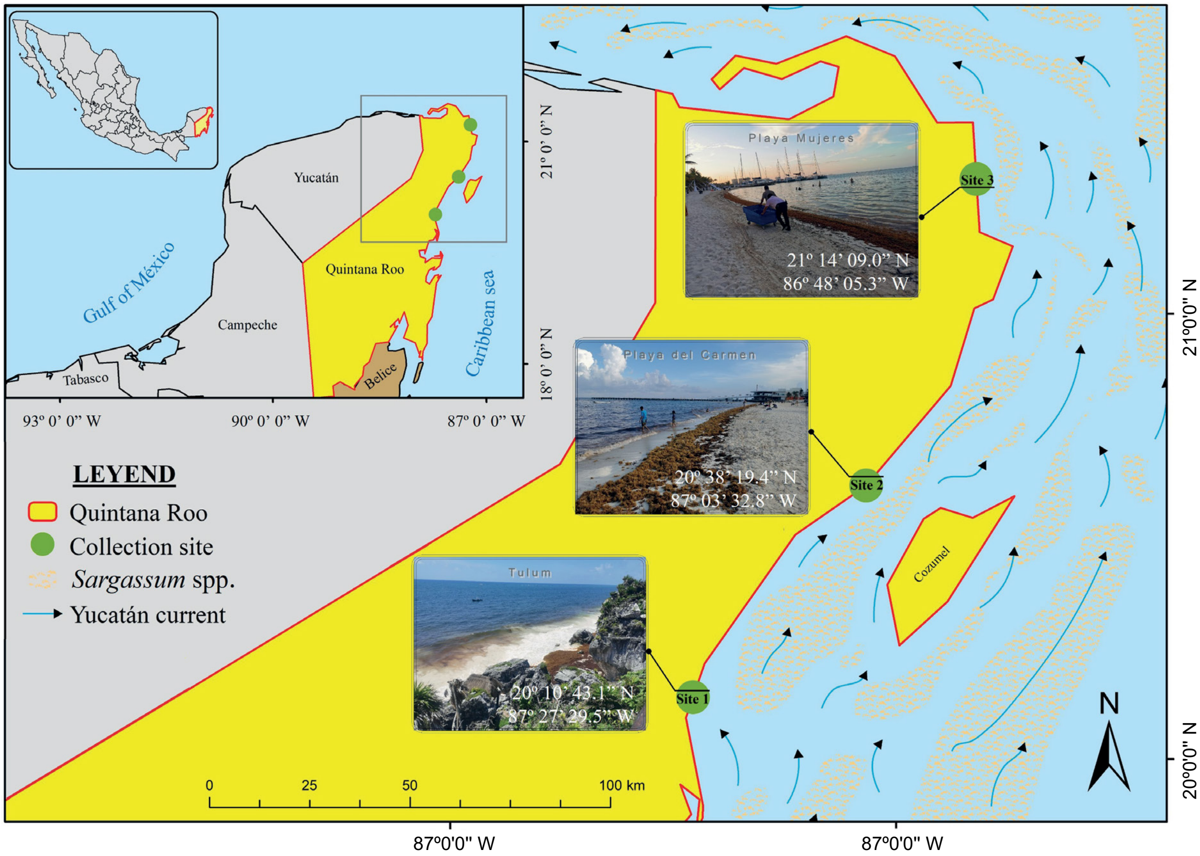

Fig. 1. Collection site of *Sargassum* biomass.

TABLE I. CONDITIONS OF *Sargassum* PRETREATMENTS PROTOCOLS.

Sample	Washing	Drying	t (h)	T (ºC)	R.H. (%)
Control	Seawater	Shadow	6	28-36	74-83
Wash-dry	Distilled water*	Solar	36	26-38	74-82
Sargassum fluitans III	Distilled water*	-	-	28-36	-
Sargassum natans I					
Sargassum natans VIII					
Phyllodes	Distilled water*	-	-	28-36	-
Thallus					
Aerocysts					

t: time; T: temperature R.H: relative humidity. *absence of chloride.

monitoring of ambient temperature and relative humidity using portable sensors. The recorded values during the drying period were consistent with official meteorological data for the corresponding dates and geographic location. During nocturnal hours, the treated samples were stored in hermetically sealed containers at ambient temperature to prevent moisture reabsorption.

Proximate analysis

American Society for Testing and Materials (ASTM) procedures were applied to quantify the moisture (MS), volatile matter (VM), ash (ASH), and fixed carbon (FC) contents in the collected biomass (ASTM 2015). The typical procedure involved placing approximately 1 g of *Sargassum* in ceramic crucibles (n = 5) until constant mass was achieved (110 ºC for 24 h) and heating them at a rate of 10 ºC/min to specific temperatures for designated times: 110 ºC for 1 h for MS, 550 ºC for 2 h for ASH, and 950 ºC for 7 min for VM. At the end of the procedure, the final mass was recorded, and the data were analyzed using equations 1 and 2, where P (%) represents the mass percentage of MS, ASH, or VM; A is the initial weight of the sample; and B is the final weight.

$$P(\%) = (A - B)/A \times 100 \tag{1}$$

$$FC(\%) = 100 - (MS\% + ASH\% + VM\%) \tag{2}$$

Ultimate analysis

Ultimate analysis was conducted using a Thermo Scientific FLASH 2000 apparatus for the quantification of carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O). Tin crucibles were employed for the determination of C, H, N, and S, while silver crucibles were used for the analysis of O. A mixture (ratio = 9:1) of *Sargassum* and a vanadium pentoxide catalyst was placed in the respective crucibles. All tests were performed three times, and the reported results represent the average values. Ultra-high purity (UHP) oxygen was used for combustion at 900 ºC, while helium (UHP) served as the reference gas.

Fourier transform infrared spectrometry (FTIR) analysis

The qualitative chemical composition of *Sargassum* was analyzed under both Control and Wash-dry conditions to identify post-treatment chemical variations. FTIR analysis was conducted using a PerkinElmer FT-IR Frontier Spectrometer, operating within an infrared range of 4000 to 500 cm^{-1}, with a resolution of 16 cm^{-1} and 64 accumulations.

Statistical analysis

Data from the ultimate and proximate analyses of both Control and Wash-dry samples were subjected to normality testing (Shapiro-Wilk) and analysis of variance (ANOVA) to assess statistically significant differences between biomass pretreatments at a 95% confidence level.

RESULTS AND DISCUSSIONS

Composition and distribution of collected *Sargassum*

Figure 2 displays representative specimens of *Sargassum* collected in this study. Collected biomass

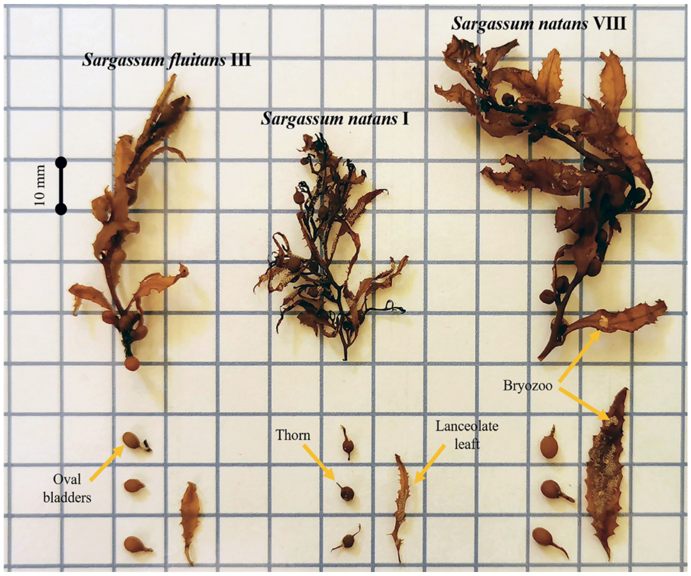

Fig. 2. Representative specimens of collected *Sargassum*.

comprises specifically *Sargassum fluitans* (Børgesen) Børgesen 1914 morphotype III and *Sargassum natans* (Linnaeus) Gaillon 1828 morphotypes I and VIII. These morphotypes correspond to *Sargassum fluitans* var. *fluitans*, *Sargassum natans* var. *natans*, and *Sargassum natans* var. *wingei*, respectively, as defined by integrative morphological analysis (Siuda et al. 2024). All three morphotypes exhibit the characteristic brown pigmentation of Phaeophyceae macroalgae, with a tree-like thallus architecture, lanceolate and serrated phyllodes, and buoyant aerocysts. Morphological differentiation is evident in several traits. The phyllodes are lanceolate and serrated across all morphotypes but vary in shape: morphotype I presents elongated and narrow phyllodes, morphotype VIII displays elongated and robust ones, while morphotype III features short and robust phyllodes. Aerocysts are oval in *Sargassum fluitans* and spherical in *Sargassum natans*, with spicules observed only in the aerocysts of morphotype I. A key diagnostic feature is the presence of spicules, small spine-like projections, on the thallus of morphotypes I and VIII, which are absent in morphotype III. These structures have previously been reported as reliable morphological markers for field identification (López et al. 2021). In addition to morphological traits, a high abundance of epibiotic bryozoans was observed encrusting the *Sargassum* thalli. Taxonomic identification confirmed the presence of cheilostome bryozoans, consistent with their known affinity for pelagic macroalgae in Atlantic ecosystems (Queiroz 2020). These colonial invertebrates preferentially settle on complex algal substrates, benefiting from elevated exposure to light and nutrient-rich microenvironments (Koehl and Daniel 2022).

Table II shows the relative abundance (%) of morphotypes across sampling sites. The compositional analysis revealed a dominant presence of *Sargassum natans* III, averaging 88.3% across all locations. In contrast, *Sargassum fluitans* VIII was minimally represented, with an average of just 1.6%. These morphotype distributions are consistent with historical data reported for the Quintana Roo coastline between 2016 and 2020, where morphotype III typically exceeded 60%, morphotype I accounted for over 23%, and morphotype VIII remained below 3% (García-Sánchez et al. 2020). The slight variation observed in the current dataset suggests that the collected biomass remains representative of the Caribbean influx. During field sampling, all three sites exhibited a continuous belt of *Sargassum* parallel to the shoreline, with visibly decaying biomass and increased water turbidity extending up to approximately 5 m offshore, as observed in **figure 1**. Sites 1 and 2, both public beaches, recorded an average accumulation height of 50 cm/m², attributed to limited cleanup efforts. In contrast, site 3, located adjacent to hotel properties with regular beach maintenance, showed significantly lower biomass accumulation, averaging just 10 cm/m². These observations underscore the influence of human intervention on the influx of coastal *Sargassum*.

TABLE II. RELATIVE ABUNDANCE (%) OF MORPHOTYPES ACROSS SAMPLING SITES

	Sargassum natans III (%)	*Sargassum fluitans* I (%)	*Sargassum fluitans* VIII (%)
Site 1	90	8	2
Site 2	95	4	1
Site 3	80	18	2
Average	88.3	10	1.6

Proximate analysis of *Sargassum*

Table III presents the proximate analysis of *Sargassum* biomass subjected to two pretreatment conditions (Control vs. Wash-dry), three morphotypes (*Sargassum natans* I, *Sargassum natans* VIII, and *Sargassum fluitans* III), and three morphological sections (aerocysts, phylloids, and thallus). Moisture content (MS) was significantly higher in the Control sample (57.9%) compared to the Wash-dry sample (18.1%).This result highlights the success of the solar drying protocol implemented in this study, which achieved substantial moisture reduction using a renewable energy source. It has been reported that the use of solar energy not only aligns with sustainable biomass processing practices but also reduces operational costs and environmental impact (Golberg et al. 2021). These findings are also consistent with previous studies emphasizing the critical role of drying protocols in biomass stabilization and downstream valorization (Milledge and Harvey 2016). For ASH content, the Wash-dry sample showed elevated levels (21.9%) relative to the Control (17.9%), suggesting a concentration of inorganic residues following *Sargassum* pretreatment. This finding underscores that while washing and solar drying can effectively reduce soluble salts, less soluble compounds, such as calcium carbonate, may persist post-treatment. Such a residual presence is consistent with previous studies, in which calcium carbonate and other mineral elements have been identified in dried *Sargassum*

TABLE III. PROXIMATE ANALYSIS OF *Sargassum* (% DRY WEIGHT).

Sample	MS (%)	ASH (%)	VM (%)	FC (%)
Control	57.9 ± 2.9*	17.9 ± 0.3*	20.7 ± 0.4*	3.5 ± 0.3*
Wash-dry	18.1 ± 0.4*	21.9 ± 0.4*	59.4 ± 0.3*	0.9 ± 1.7*
Sargassum natans I	72.4 ± 3.2	6.6 ± 2.3	17.0 ± 0.5	3.9 ± 0.4
Sargassum natans VIII	67.7 ± 4.6	4.8 ± 0.5	21.6 ± 0.5	5.8 ± 0.3
Sargassum fluitans III	67.4 ± 4.7	6.5 ± 0.9	19.3 ± 0.3	6.8 ± 0.8
Aerocysts	72.9 ± 5.2	5.5 ± 0.6	19.0 ± 0.3	6.8 ± 0.3
Phylloids	62.0 ± 4.8	8.4 ± 0.9	29.5 ± 2.2	1.8 ± 0.6
Thallus	56.6 ± 6.9	7.0 ± 1.2	32.6 ± 3.9	3.8 ± 0.5

Note: (*) indicate statistically significant differences ($p < 0.05$). MS: moisture; ASH: ash; VM: volatile matter; FC: fixed carbon.

tissues collected along the Mexican Caribbean coast (Rodríguez-Martínez et al. 2020). These results highlight the importance of understanding the mineral composition of *Sargassum* biomass after deposition and accumulation along coastal environments, particularly for its valorization in bioenergy applications. VM was markedly higher in the Wash-dry sample (59.4%) compared to the Control (20.7%), likely due to the effects of solar drying. This increase is consistent with thermal degradation patterns observed in brown macroalgae, in which dehydration enhances the concentration of thermolabile organic compounds (Torabi et al. 2021). A higher VM proportion is advantageous for the thermochemical valorization of marine biomass, as it indicates greater availability of volatile compounds that can be thermally degraded to biogas and bio-oil. These compounds, mainly low-molecular-weight carbohydrates, lipids, and phenolics, are key precursors in pyrolysis and gasification processes, contributing to energy yield and product quality (Petersen et al. 2024). Therefore, the elevated VM content in the wash-dry sample suggests improved suitability of the processed *Sargassum* for bioenergy applications. Interestingly, FC content was significantly lower in the Wash-dry sample (0.9%) than in the Control (3.5%), a result that initially appears counterintuitive given the concurrent increase in ash content. However, this discrepancy can be explained by the redistribution and volatilization of organic compounds during solar drying. In marine macroalgae such as *Sargassum*, the FC fraction is derived from non-volatile organic matter, whereas ash represents total inorganic residue (Martínez-Meraz et al. 2023). The drying process may promote the loss of soluble organic constituents that would otherwise contribute to FC, without proportionally affecting ash accumulation. Similar trends have been reported in *Sargassum fusiforme*, in which drying methods significantly influenced the retention of carbonaceous fractions and volatile profiles (Song et al. 2024). Therefore, the observed reduction in FC does not contradict the increase in ash but rather reflects the selective depletion of organic carbon during pretreatment. These findings underscore the importance of understanding the physical and chemical dynamics of biomass during solar drying, particularly when evaluating its suitability for biochar production. While elevated VM enhances potential for bioenergy conversion, reduced FC may limit char yield, necessitating optimization of drying protocols depending on the intended valorization pathway.

Table III also presents the proximate analysis of *Sargassum* categorized by morphotype (*I*, *VIII*, and *III*) and by morphological section (aerocysts, phylloids, and thallus). Among morphotypes, *Sargassum natans I* exhibited the highest MS content (72.4%), followed by *Sargassum natans VIII* (67.7%) and *Sargassum fluitans III* (67.4%). These variations may reflect morphotype-specific structural adaptations, as discussed by another research (Schell et al. 2015). Ash content was lowest in *Sargassum natans VIII* (4.8%), while *Sargassum natans I* and *Sargassum fluitans III* showed comparable values (~6.5%), suggesting differential mineral uptake or epiphytic colonization (Koehl and Daniel 2022). VM and FC were highest in morphotype *VIII* (VM: 21.6%, FC: 5.8%), indicating a richer organic matrix potentially linked to its ecological niche and photosynthetic activity (Hu et al. 2021). Regarding morphological sections, aerocysts showed the highest MS (72.9%) and elevated VM (19.0%), consistent with their buoyant structure and low ash content (5.5%), supporting their role in flotation rather than nutrient storage (Schell et al. 2024). Phylloids exhibited high VM (29.5%), intermediate MS (62.0%), and ASH content (8.4%), while FC was minimal (1.8%), reflecting active photosynthetic tissues with limited lignocellulosic density (Owusu et al. 2024). Thallus sections showed the lowest MS (56.6%) and highest VM (32.6%), indicating dense structural biomass. Their FC content (3.8%) exceeded that of phylloids, consistent with their supportive function and potential suitability for biochar production (Martínez-Meraz et al. 2023). Although compositional heterogeneity was observed across morphotypes and structural sections, statistical analysis revealed no significant differences in proximal composition between morphotypes or between morphological sections. This finding indicates that prior segregation of *Sargassum* biomass by morphotype or structure is not necessary for its valorization. Consequently, the discussion will now focus on the influence of pretreatment strategies (specifically the Wash-dry protocol) on biomass composition.

Ultimate analysis of *Sargassum* biomass

Table IV presents the ultimate analysis of C, H, O, N, S, and the HHV for both untreated (Control) and pretreated (Wash-dry) *Sargassum* samples. The elemental composition of both samples aligns with previously reported values for *Sargassum* collected from Guadalupe Island (Milledge and Harvey, 2016) and the Mexican Caribbean (Saldarriaga-Hernández et al. 2020), which suggest compositional consistency across geographic and temporal scales. Statistical analysis confirmed no significant differences ($p < 0.05$) in CHONS content between the two evaluated samples, indicating that the washing and solar drying process did not alter the intrinsic chemical makeup of the *Sargassum*. The HHV values for both samples (14.8 MJ/kg for Control and 14.2 MJ/kg for Wash-dry) were also statistically indistinguishable and comparable to values reported for *Sargassum* collected along the Quintana Roo coast (Saldarriaga-Hernaádez et al. 2021). These findings suggest that the applied pretreatment effectively removed surface contaminants without compromising the energy potential of the *Sargassum*. This is particularly relevant for thermochemical recovery, such as pyrolysis or gasification, where maintaining the calorific integrity of biomass is essential (Datta et al. 2024). The preservation of HHV further supports the feasibility of using pretreated *Sargassum* as a renewable feedstock for bioenergy applications, even after environmental cleaning protocols. Although the numerical differences in C, S, and HHV between Control and Wash-dry samples appear minimal, their statistical significance is supported by low standard deviations and precise analytical resolution. The slight increase in C content following solar drying may reflect the relative concentration of organic matter due to moisture loss, without substantial alteration of the structural carbon of the biomass. Similarly, the marginal reduction in S content likely results from the partial removal of soluble sulfur compounds during washing, while more stable sulfur fractions remain unaffected. In the case of HHV, the small decrease observed in the Wash-dry sample is attributable to the interplay between reduced hydrogen content and increased ash fraction, both of which influence calorific value, as highlighted previously on **table III**. These subtle shifts, though numerically modest, were consistently detected across replicates and validated through ANOVA, underscoring the sensitivity of ultimate analysis and the importance of rigorous statistical treatment when evaluating biomass pretreatment effects.

TABLE IV. ULTIMATE ANALYSIS AND HIGHER HEATING VALUE (HHV) OF CONTROL AND WASH-DRY SAMPLES

Parameter	Control (% wt)	Wash-Dry (% wt)	p value
Carbon (C)	30.6 ± 0.4	31.5 ± 0.3	0.041
Hydrogen (H)	4.6 ± 0.2	3.4 ± 0.1	0.008
Oxygen (O)	17.2 ± 0.3	13.6 ± 0.2	0.015
Nitrogen (N)	2.9 ± 0.1	1.7 ± 0.1	0.003
Sulfur (S)	1.5 ± 0.1	1.4 ± 0.1	0.049
HHV (MJ/kg)	14.8 ± 0.2	14.2 ± 0.2	0.047

Note: Statistical significance was assessed using one-way ANOVA with $\alpha = 0.05$. All comparisons between Control and Wash-dry treatments yielded statistically significant differences.

Fourier transform infrared analysis of *Sargassum* biomass

Figure 3 presents the FTIR spectra of Control and Wash-dry samples to determine whether the pre-treatment influenced the presence of compounds relevant for thermochemical recovery. The FTIR analysis revealed a diverse array of functional groups, including hydroxyl (O–H), carbonyl (C=O), amide (N–H), and ether (C–O) vibrations, which are indicative of polysaccharides such as alginate ($C_6H_7O_6Na$) and fucoidan ($C_6H_9O_3SO_3$), as well as proteinaceous and lipidic components (Peniche-Pavía et al. 2024). Signals near 3400 cm^{-1} and 1050 cm^{-1} confirmed the presence of hydroxyl-rich polysaccharides, while bands around 1740 cm^{-1} and 1550 cm^{-1} correspond to esterified phenolics and amide groups, respectively (Tagliapietra and

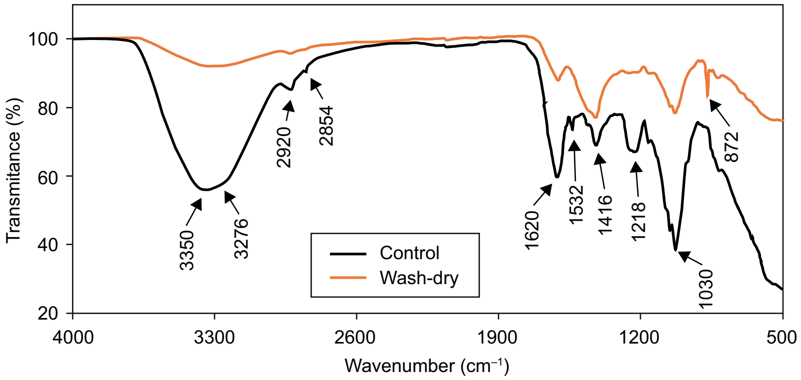

Fig. 3. Fourier transform infrared spectra of *Sargassum* samples (Control and Wash-dry).

Clerici 2023), suggesting a complex biochemical matrix. Inorganic residues such as calcium carbonate (~875 cm^{-1}), siliceous sand (~780 to 800 cm^{-1}), and marine salts (~615 to 670 cm^{-1}) (Tulashie et al. 2024) were also detected in both samples. Finally, these spectral findings align with the results of the proximal (**Table III**) and elemental (**Table IV**) analyses, which showed high VM content (up to 59.4%), moderate ash levels (17.9 to 21.9%), and significant C, H, N, and O contents, supporting potential for bioenergy applications. The removal of salts and mineral debris may improve the quality of the biomass for thermochemical conversion, as it potentially reduces inorganic interference and enhances energy yield. Together, these analytical approaches provide a comprehensive understanding of *Sargassum* composition and its suitability for valorization strategies. Although the FTIR spectral bands identified in both Control and Wash-dry samples confirm the presence of key functional groups associated with marine polysaccharides, proteins, and mineral residues, a noticeable attenuation in band intensity was observed in the Wash-dry treatment, particularly around 3400 cm^{-1} (O–H stretching) and 1740 cm^{-1} (C=O stretching). This reduction does not indicate a loss of structural integrity but rather reflects the selective removal of soluble organic compounds, such as free phenolics, carboxylic acids, and low-molecular-weight esters, which contribute to spectral signal enhancement. Washing with distilled water followed by solar drying likely removed surface-bound and loosely associated compounds that enhance vibrational responses, resulting in cleaner but less intense spectra. Similar trends have been reported in marine macroalgae subjected to aqueous pretreatments, where FTIR signal attenuation was attributed to the depletion of interfering solutes and non-structural organics (Yamin et al. 2017). Moreover, the increase in carbonate signal (~875 cm^{-1}) and the disappearance of siliceous and saline bands (780 to 800 and 615 to 670 cm^{-1}) further support the hypothesis of selective purification rather than analytical error. Nonetheless, given the subtle nature of these spectral shifts, future analyses using higher-resolution FTIR or complementary techniques, such as Raman spectroscopy, are recommended to validate these observations and to rule out potential overlap or baseline distortions.

Table V shows the list of relative compounds identified in Control and Wash-dry samples for comparative purposes. The FTIR spectral analysis reveals a significant reduction in mineral interference and a clearer organic profile in *Sargassum*. The detection of characteristic absorption bands corresponding to calcium carbonate ($CaCO_3$) and silicon dioxide (SiO_2) in the FTIR spectra is consistent with the presence of calcareous epibiotic residues and siliceous particulates adhered to the biomass surface, respectively. The most intense signal associated with calcium carbonate, observed in the Wash-dry sample near 875 cm^{-1}, indicates minimal removal of epibiotic bryozoans previously identified on the *Sargassum* surface (**Fig. 2**). This persistence is likely attributed to the low solubility of calcium carbonate in water, which limits its detachment during aqueous washing (Richardson and Melchers 2024). On the other hand, the solar drying step, performed under controlled exposure, preserved the integrity of organic functional groups while reducing moisture content, as evidenced by the decreased intensity of the broad O–H stretching

TABLE V. FOURIER TRANSFORM INFRARED SPECTRAL BANDS IDENTIFIED IN CONTROL AND WASH-DRY SAMPLES

(cm^{-1})t	Functional Group/Vibration	Associated Compound	Intensity (Control)	Intensity (Wash-dry)	Relative compound	Effect of pretreatment
~3400	O–H stretching	Hydroxyl (water, alcohols)	Strong	Medium	Fucoidan, alginic acid	Intrinsic moisture and polysaccharides retained
~2920	C–H stretching	Alkanes (lipids)	Medium	Medium	Phytol, palmitic acid	Lipid structures remain stable
~1740	C=O stretching	Carbonyl (esters, acids)	Medium	Weak	Esterified phenolics	Surface-bound esters removed
~1650	C=C stretching/Amide I	Alkenes/Proteins	Medium	Medium	Proteins, carotenoids	Proteinaceous structures preserved
~1550	N–H bending	Amide II	Medium	Medium	Amino acids, peptides	Minimal nitrogen loss
~1450	C–H bending	Alkanes	Medium	Medium	Hydrocarbons, wax esters	Aliphatic chains remain intact
~1250	C–O stretching	Esters/Ethers	Medium	Medium	Fucoidan, alginate derivatives	Ether-linked polysaccharides resist washing
~1050	C–O stretching	Alcohols/Polysaccharides	Medium	Medium	Mannitol, laminarin	Carbohydrate-rich matrix preserved
~875	Out-of-plane CO_3^{2-} bending	Carbonate ($CaCO_3$)	Weak–Medium	Strong	Calcium carbonate	Minimal elimination of calcareous debris
~780-800	Si–O symmetric stretching	Silica (SiO_2, sand)	Weak	Absent	Quartz, siliceous sand	Siliceous particles washed out
~615-670	SO_4^{2-} bending/Cl^- traces	Marine salts (salinity)	Weak	Absent	NaCl, $MgSO_4$	Desalination confirmed

band at ~3400 cm^{-1} (**Fig. 3**). This correlates with the moisture reduction observed in the proximate analysis (**Table III**), where the Wash-dry sample exhibited lower moisture levels compared to the untreated biomass. Furthermore, the FTIR signals associated with polysaccharides (C–O–C and C–OH vibrations at 1050 to 1150 cm^{-1}), lipids (C–H stretching at ~2920 and ~2850 cm^{-1}), and protein-related amide bands (~1650 cm^{-1}) (Peniche-Pavía et al. 2024) were more discernible in the Wash-dry sample. These findings are supported by the proximate analysis (**Table III**) and elemental composition (**Table IV**), which showed increased relative proportions of VM and organic content due to the removal of inorganic ballast. The reduction in ash content and the stabilization of nitrogen levels for the Wash-dry sample suggest the pretreatment preserved nitrogenous compounds, potentially linked to protein fractions detected in the FTIR spectrum. Collectively, these results confirm that washing and solar drying pretreatments enhance the spectral clarity of FTIR analysis and improve the compositional quality of *Sargassum*. This integrated approach supports its suitability for bioenergy conversion, biopolymer extraction, and nutrient recovery, while ensuring reproducibility and minimizing interference from mineral components.

Limitations of the study

Although this study provides valuable insights into the physical, chemical, and spectroscopic effects of washing and solar drying on *Sargassum* biomass, certain limitations must be acknowledged. Nonetheless, the findings may contribute meaningfully to the expanding body of research on sustainable *Sargassum* valorization and offer a reproducible framework for biomass conditioning in bioenergy and bioproduct applications.

The FTIR analysis was conducted using mid-resolution spectra, which may not fully capture subtle vibrational changes in functional groups, particularly in samples with low organic content or overlapping bands. Future studies employing high-resolution FTIR or complementary techniques such as Raman spectroscopy or nuclear magnetic resonance (NMR) could enhance spectral discrimination and confirm the observed trends.

Although statistical significance was achieved for parameters with marginal numerical differences (e.g., C, S, and HHV), the interpretation of these results requires caution. The sensitivity of elemental analysis and calorimetry to minor compositional shifts underscores the need for replication across broader sample sets and seasonal conditions to validate consistency and reduce potential analytical bias.

This study employed a single pretreatment protocol: washing with distilled water followed by solar drying, which represents a low-energy-intensity approach. However, it did not investigate alternative drying techniques (e.g., oven-drying, freeze-drying) or washing agents (e.g., acidified water, chelating solutions), which could yield different outcomes regarding contaminant removal and biomass integrity. Comparative evaluations of diverse pretreatment strategies would enhance the generalizability and applicability of the findings.

The ecological variability of *Sargassum*, including morphotype composition, epiphytic load, and environmental exposure, may influence biomass characteristics beyond the scope of this study. Although efforts were made to standardize collection and processing, future work should incorporate broader geographic sampling and temporal replication to account for natural heterogeneity.

CONCLUSIONS

This study investigated the effects of pretreatment on the composition and properties of *Sargassum* collected from the Mexican Caribbean. The findings demonstrate that the pretreatment, specifically washing and solar drying, did not significantly alter the chemical composition or the presence of organic compounds. Characterization of *Sargassum* through FTIR spectroscopy, proximate analysis, and elemental analysis revealed that the Wash-dry pretreatment reduced surface-bound inorganic contaminants such as marine salts, siliceous sand, and loosely attached calcium carbonate. FTIR spectra confirmed the preservation of key organic functional groups, including hydroxyl, carbonyl, and polysaccharide-associated vibrations, indicating that the biochemical integrity of the biomass was maintained. Proximate analysis showed a reduction in moisture and ash content, while volatile matter and fixed carbon remained stable or improved, reflecting enhanced organic concentration. Elemental analysis further demonstrated consistent CHONS profiles between untreated and pretreated samples, confirming that the internal chemical composition was unaffected. Collectively, these results validate the Wash-dry protocol as a selective and non-destructive method for improving *Sargassum* quality. The scientific contribution of this study lies in demonstrating that *Sargassum* can be valorized without prior segregation by morphotype or structural section, and that pretreatment enhances its suitability for bioenergy applications.

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